

ARMY RESEARCH LABORATORY



# "Clean Burning" Low Flame Temperature Solid Gun Propellants

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## 1. INTRODUCTION

Incomplete combustion of solid gun propellants is a common occurrence during the firing of both experimental and fielded gun systems. Included among the products of this phenomenon are nitrogen oxides (NO<sub>x</sub>) and carbon monoxide (CO). CO poses the most serious health hazard (Weyandt and Ridgeley 1993), and can be found at such dangerously high levels that health agencies may impose restrictions on the number of test firings that can be performed per day. Unfortunately, elimination of CO from gun propellant combustion products is not feasible from a ballistic standpoint. However, the corrosive nature of nitrogen oxides (NO<sub>x</sub>'s where  $x = 1,2$ ) also make them a matter of concern for both users and environmental and health-related agencies. Snelson et al. (1989) measured breech NO<sub>x</sub> concentrations that were  $10^5$  times greater than predicted by thermochemical calculations, indicating that the actual problem may be more serious than suggested by equilibrium calculations. While it may be argued that bore evacuators protect tank crews against toxic combustion products, this is true only when the evacuators are functioning properly and are intact. It has been reported that a hole as small as 2 cm is sufficient to allow dangerously high levels of toxic gases into the crew compartment. It has been suggested that even intact evacuator systems do not keep tank compartments free of propellant combustion gases. Exposure of test crews to combustion gases is becoming increasingly more common as test site personnel resort to enclosed (muffled) bays to reduce noise levels near urban populations. At such sites, large fans must ventilate the test bay for several minutes to remove toxic vapors before workers can enter the test area.

Solid residues resulting from incomplete combustion also pose significant safety and environmental concerns. For example, smoldering residues remaining after firing often result in significant quantities of smoke. This low pressure pyrolysis generates much larger quantities of NO<sub>x</sub> and other nonequilibrium gases than does high pressure combustion. Re-ignition of propellant residue in fielded tanks has been reported (Wren 1993). Propellant residue in breeches is also said to be responsible for the creation of sealing problems (Rinaldi 1993).

As discussed above, incomplete combustion of solid propellants can pose a significant hazard to the health and safety of military and civilian personnel. The work presented here is intended to address and reduce this hazard by identifying solid propellant formulations that will burn more cleanly and more completely than current formulations.

## 2. BACKGROUND

It has been known for some time that M30 gun propellant (composed of nitrocellulose, nitroguanidine and nitroglycerine) differs from other NC-based propellant in that its flame lacks a "dark zone" (i.e., it does not have a nonluminous zone) (Vanderhoff 1988, 1989, 1991; Vanderhoff, Teague, and Kotlar 1992; Teague, Singh, and Vanderhoff 1993). Dark zone chemistry is dominated by the chemistry of NO, specifically, by the slow conversion of NO to  $N_2$  (Vanderhoff, Teague, and Kotlar 1991). Much attention has been given to this chemistry because of its potential importance to delayed ignition (Vanderhoff, Teague, and Kotlar 1991, 1992). It was proposed by Anderson (1987) that the absence of the dark zone in M30 might suggest that nitroguanidine was behaving as a "De-NOx agent" (i.e., a species capable of reducing NOx to  $N_2$ ).

A proposed mechanism for the removal of NO and, hence, NOx's, by amines is described in Figure 1 (Miller and Bowman 1989). The essence of this process is the consumption of NO in reaction paths that lead ultimately to  $N_2$ . Note that  $NH_3$  does not react with NO (the reaction is endothermic by 60 kcal/mole).

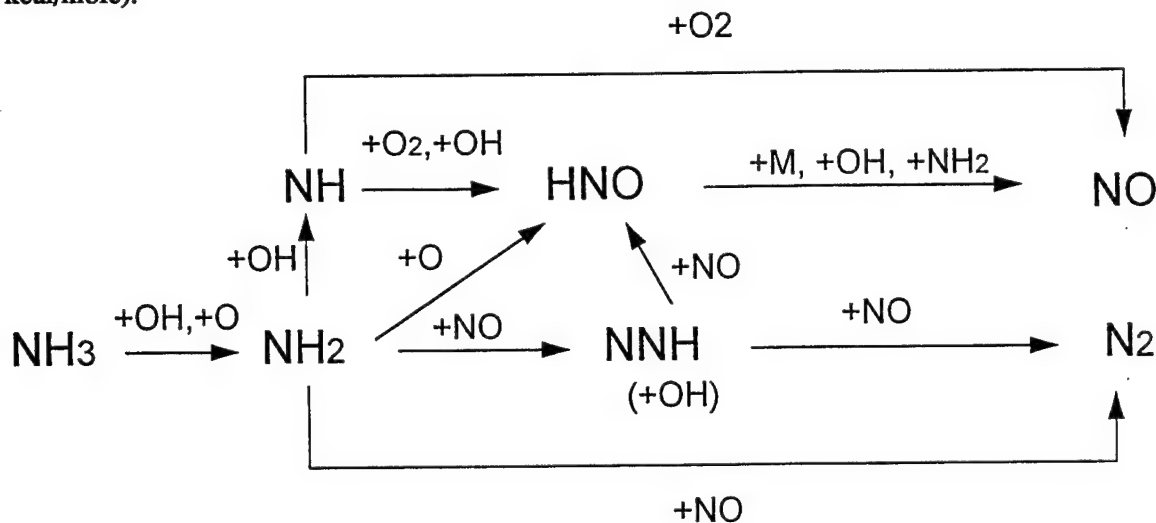


Figure 1. The Miller and Bowman thermal De-NOx mechanism.

De-NOx agents have been shown to be effective in eliminating NOx production in internal combustion engines and other devices. In these applications, the agents are ammonia- or amine-producing species that reduce NO to  $N_2$ . In nitrate ester- or nitramine-based solid gun and rocket propellants, NO is produced from the  $-NO_2$  groups in the energetic oxidizer (e.g., RDX), polymer (e.g., nitrocellulose NC), or plasticizer (e.g., nitroglycerine, NG). The concept for the current work is based on the use of De-NOX

agents in solid propellants to reduce NO to  $N_2$  at or near the burning propellant surface; the increased near-surface energy release should also lead to more complete combustion (i.e., more equilibrium products and less solid residue) and reduction of other products of a toxic or carcinogenic nature. In addition to reducing NOx levels in the combustion products of solid gun propellants, burn rate modification by De-NOx agents is also possible. Previous attempts to "catalyze" the burning of solid propellants have made use of oxidizing agents (e.g, PbO), rather than reducing agents (e.g,  $NH_3$ ). (Note: an exception to this has been found [Fifer and McBratney 1983; Fifer and Cole 1980].) If De-NOx agents can effectively eliminate NOx from the combustion products of solid propellants, they may also serve as effective burning rate "catalysts," thereby simultaneously preventing pollution (by both NOx and heavy metals) and enhancing performance.

Based on this background information, an investigation into the effects of ammonia- and amine-generating compounds on solid propellant combustion and pyrolysis products was undertaken. In an examination of the pyrolysis products of M30 (a triple-base propellant) and two "nitroguanidine-free" nitrocellulose-based propellants (M9 and M10, double- and single-base propellants, respectively), it was observed that M30 actually generates more NO than do the propellants without nitroguanidine. These results suggest that while nitroguanidine might participate in the elimination of NO under high pressure combustion conditions, it does not do so at atmospheric pressure. Results presented in this report indicate that while nitroguanidine does generate  $NH_3$ , it also generates significant quantities of NO and is therefore an unsuitable De-NOx agent.

The specific focus of the present work is on the use of urea as a De-NOx agent, especially under pyrolysis conditions where traditional coolants such as nitroguanidine appear to be ineffective. While this application may be new, a survey of the literature reveals that use of urea as a stabilizer in solid propellants is not. In the 1980s, urea was used as a stabilizer in certain double-base propellants. The propellants were invented by Hudson Maxim in collaboration with R. D. Schüpphaus, and were known as "Maxim-Schüpphaus Powders." The propellants also have the distinction of being the first manufactured in the form of multiperforated grains. Formulations typically contained 0.5–1.0 wt-% urea (Kaye 1978).

### 3. EXPERIMENTAL

3.1 Samples. Experimental quantities of a double-base, JA2-like solid propellant were fabricated by handmixing acetone solutions of JA2 with ground urea. Pyrolysis samples were prepared by casting films

of the handmixed propellants on aluminum foil. Candidate De-NO<sub>x</sub> agents included triaminoguanidine nitrate (TAGN), nitroguanidine (NQ), triamino-trinitro-benzene (TATB), ammonium carbonate, and urea. Structures of these compounds are given in Figure 2.

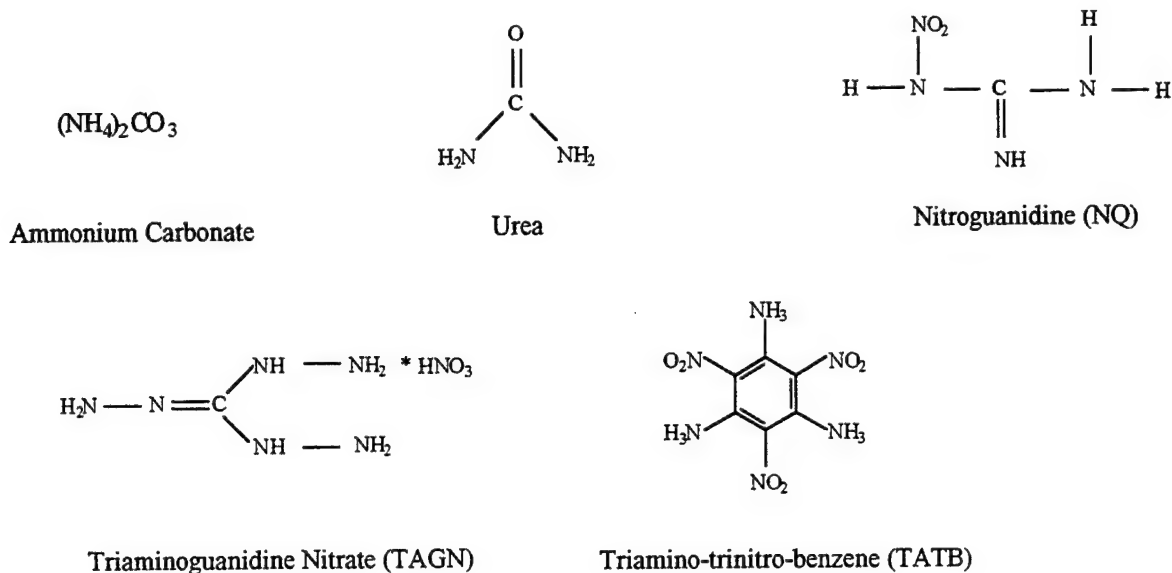


Figure 2. Structure of candidate De-NO<sub>x</sub> agents.

3.2 Chromatographic Instrumentation. Analyses of pyrolysis products were conducted using a Hewlett Packard 5965 gas chromatograph (GC) interfaced to a Hewlett Packard Model 5965A infrared detector (IRD), and 5970 mass spectrometric detector (MSD). Only MS results are reported here. Chromatographic conditions: Quadrex capillary column 0.32-mm  $\times$  25-m  $\times$  3- $\mu$ m OV-17 film; oven program: 50° C for 3 min, then 50° C to 200° C at 10°/min; injector and interface chamber held at 200° C.

3.3 Pyrolysis Experiments. Samples were placed in quartz tubes and held in place with plugs of glass wool. Tubes were inserted into a coil-type Pyroprobe (Chemical Data Systems [CDS], Model 122) that was connected via a heated CDS interface chamber to the splitless injector of the GC. The heated GC interface was continuously swept with helium carrier gas. Approximately 0.6 mg of sample was pyrolyzed for each analysis. All samples were pulse-pyrolyzed at 1,000° C.

3.4 Thermal Analysis. Differential scanning calorimetry (DSC) was performed using a Mettler DSC 30 calorimeter interfaced to a Mettler TC10A processor. The heating rate was 10° C/min. Samples were

placed in crimped aluminum pans with three pin holes punched through the cover. Analyses were run under a nitrogen atmosphere (20 mL/min).

3.5 Calculations. Blake (Freedman 1981) calculations were performed using a value of  $-80.227$  kcal/g-mol for the heat of formation of urea, and "standard" library values for JA2 propellant.

#### 4. RESULTS AND DISCUSSION

Screening runs of potential De-NO<sub>x</sub> agents were performed by pyrolyzing the materials at 1,000° C. The primary basis for acceptance during screening runs was that the materials thermally decompose to generate ammonia or amines. Results of screening tests, as well as the melting (or decomposition) temperatures for the candidate De-NO<sub>x</sub> agents, are given in Table 1. Based on the information in Table 1, it was concluded that the energetic materials (TAGN, NQ, and TATB) were unsuitable as De-NO<sub>x</sub> agents because they generate significant quantities of NO. Ammonium carbonate was eliminated from further consideration because of its low decomposition temperature and hygroscopicity.

Table 1. Relative Pyrolysis Yields and Melting or Decomposition Temperatures for Several Candidate NO<sub>x</sub> Agents

Compound	Relative Pyrolysis Yield		Temperature (°C)	Comments
	NH <sub>3</sub> <sup>a</sup>	NO <sup>a</sup>		
Ammonium Carbonate	329	0	58 <sup>b</sup>	good De-NO <sub>x</sub> agent; hygroscopic
Urea	101 <sup>c</sup>	0	133	good thermal stability; compatible with NC
TAGN	281	111	216 <sup>b</sup>	generates NO on pyrolysis
NQ	66	194	225–250 <sup>b</sup>	generates NO on pyrolysis
TATB	33	174	325 <sup>b</sup>	generates NO on pyrolysis

<sup>a</sup> Pyrolysis at 1,000° C. Peak area determined by pyrolysis/GC-MS; sample mass = 0.3 mg.

<sup>b</sup> Decomposes.

<sup>c</sup> Urea also generated significant amounts of large pyrolysis products (e.g., vapor phase urea). It is expected that under combustion conditions, even more NH<sub>3</sub> will be generated.

Figure 1 is a scatter plot showing the relationship between the NO Peak area (in arbitrary units) and the weight percentage of urea. The y-axis, labeled 'NO Peak area (arb. units)', ranges from 0 to 200. The x-axis, labeled 'Wt-% Urea', ranges from 0 to 12. The data points show a general decrease in NO Peak area as the weight percentage of urea increases. Two data points at 5 Wt-% Urea are highlighted with error bars.

Wt-% Urea	NO Peak area (arb. units)
0	~195
1	~165
2	~130
3	~105
4	~80
5	~130 (with error bars)
5	~65
6	~50
7	~38
8	~30
9	~22
10	~15
11	~70 (with error bars)

- Calc. by Blake\*\*

**\*\* Combustion product;  
for comparison, scaled**

Figures 4 and 5 show flame temperatures and impetus, respectively, calculated using the Blake code. As expected, the impetus is observed to decrease as the percentage of urea in the formulation increases. The net change in impetus for a JA2-like propellant containing 4 weight-percent urea is approximately 4% relative to neat JA2. The flame temperature of the JA2/urea propellant containing 4 weight-percent urea is approximately 200 K lower than that of neat JA2. The decrease in projectile velocity for the same JA2/urea propellant is expected to be approximately 2% (considering that muzzle velocity is proportional to the square root of the impetus).



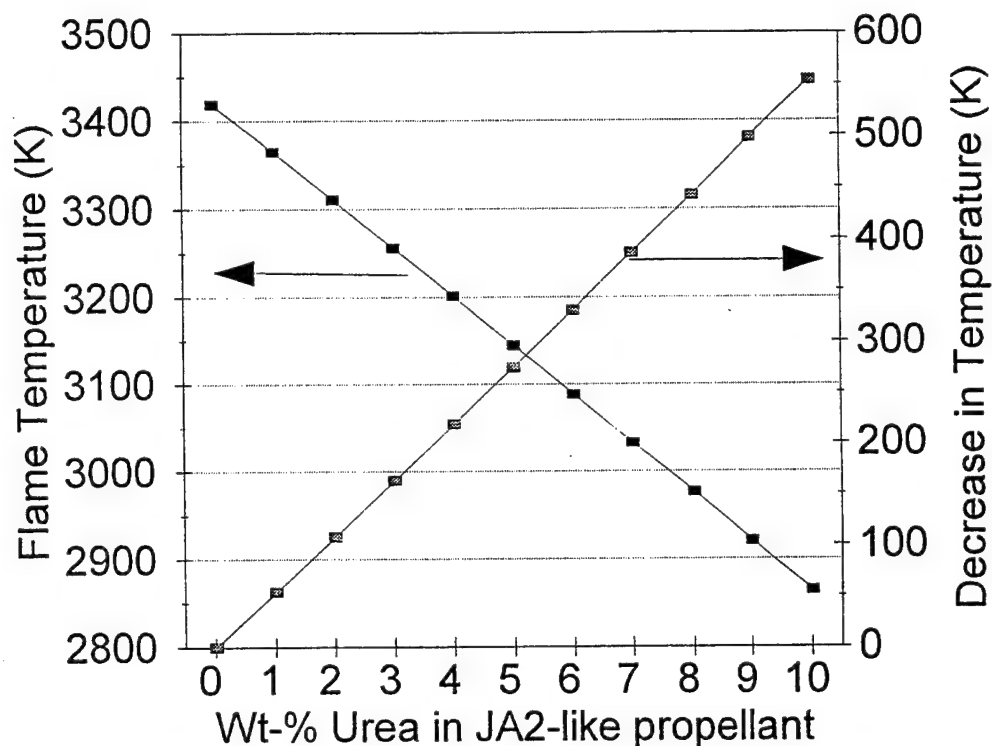


Figure 4. Calculated flame temperature for JA2/urea propellant; from Blake calculations at 0.2 g/cm<sup>3</sup> nominal gas phase density.

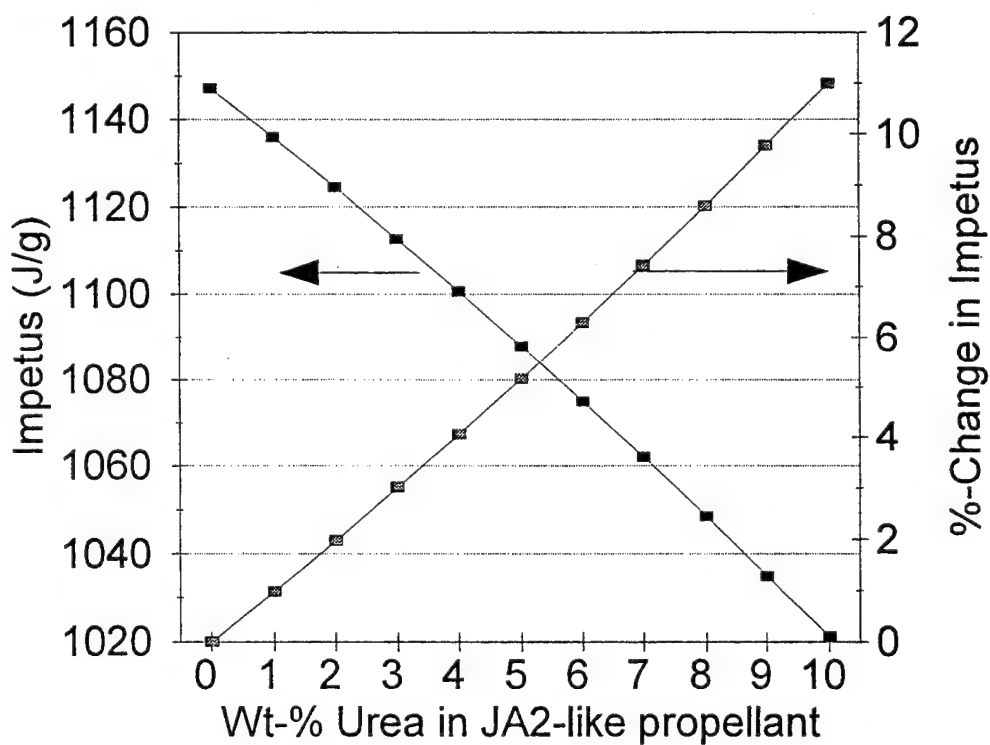


Figure 5. Calculated impetus for JA2/urea propellant; from Blake calculations at 0.2 g/cm<sup>3</sup> gas phase density.

The DSC thermograms presented in Figure 6 indicate that the JA2/urea formulation (7 weight-percent urea) decomposes at approximately the same temperature as does neat JA2. This result indicates that JA2 and urea are chemically compatible. This is to say that urea will not act as a catalyst in the thermal decomposition of JA2 at these heating rates. Physical compatibility has yet to be determined.

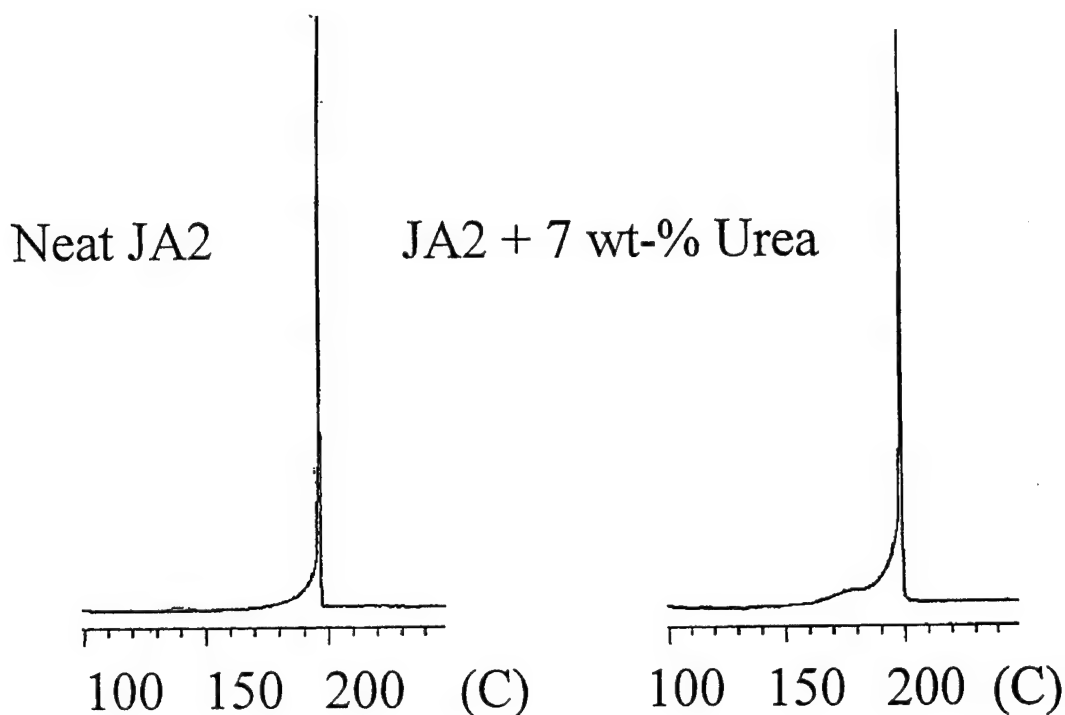


Figure 6. DSC thermograms for JA2 and JA2/urea propellant (little/no change in exotherm temperature observed, indicative of good compatibility).

## 5. CONCLUSION

Based on the results presented here, it is concluded that urea is an attractive De-NO<sub>x</sub> agent: it effectively reduces the level of NO generated by pyrolysis of JA2-like propellants, yet has little effect on performance when used at low levels of incorporation. Added benefits of the use of urea as a De-NO<sub>x</sub> agent include: (1) urea has been reported to be a successful flash suppressant (Klingenberg and Heimerl 1992); and (2) urea results in flame temperature decreases that are expected to significantly extend the life of gun barrels. Results reported for the use of polyurethane additives in double-base propellants (Huwei and Ruonong 1992) suggest a similar flame temperature suppression and concomitant wear reduction.

Combined with the reduction in the production of corrosive NOx's, the reduction of flame temperature achieved by the use of urea in solid gun propellants should extend the life of gun barrels while at the same time improving air quality at R&D test centers, as well as in training and battlefield situations.

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